BEST AVAILABLE IMAGES

Defective images within this document are accurate representations of the original documents submitted by the applicant.

Defects in the images may include (but are not limited to):

- BLACK BORDERS
- TEXT CUT OFF AT TOP, BOTTOM OR SIDES
- FADED TEXT
- ILLEGIBLE TEXT
- SKEWED/SLANTED IMAGES
- COLORED PHOTOS
- BLACK OR VERY BLACK AND WHITE DARK PHOTOS
- GRAY SCALE DOCUMENTS

IMAGES ARE BEST AVAILABLE COPY.

As rescanning documents *will not* correct images, please do not report the images to the Image Problem Mailbox.

REMARKS

The applicants are requesting continued prosecution of this application in order to obtain the Examiner's consideration of art cited in an opposition filed against the applicants' corresponding EP 1 149 149 B1. Accordingly, the applicants submit herewith copies of the following:

1. opposition;
2. opposition references (1) to (6);
3. PTO-1449 listing the references;
4. applicants' EP 1 149 149 B1 which is being opposed; and
5. applicants' reply of April 30, 2004 to the opposition.

The claims have been amended to emphasize differences over the references. As amended, the claims are thought to be allowable over the opposition references on the basis of the arguments advanced in applicants' reply to the opposition, the reply being incorporated herein by reference. Accordingly, favorable consideration of the application on the basis of the present submission is requested.

As the Examiner will appreciate, the applicants' claims 1-4 and 6-17 define the active material as one comprising a fragrance as in previously allowed claim 5 (now canceled as redundant) and claim 17.

New claims 18 and 20 are modeled after claims 1 and 11 but bring in the option where the active material may be an enzyme or a fragrance. Claims 19 and 21 depend from claims 18 and 20, respectively, and specify an enzyme as active material. Basis for the use of an enzyme as claimed is found at, for example, page 4, line 8.

As indicated, the claims, as amended, including the newly added claims, are thought to be allowable over the opposition art for the reasons noted in the applicants' reply to the opposition. Accordingly, allowance is requested.

Respectfully submitted,

MORGAN, LEWIS & BOCKIUS LLP

By: _____
Paul N. Kokulis
Reg. No. 16,773

Date: June 3, 2004

Customer No. 09629
1111 Pennsylvania Avenue, N.W.
Washington, D.C. 20004
Phone: (202) 739-3000
Facsimile: (202) 739-3001
Direct: (202) 739-5455

Notice of Opposition to a European Patent

11 Aug. 2003

for EPO use only

I. Patent opposed

Opp. No. OPPO (1)

Patent No. 1 149 149

Application No. 00901233.7

Date of mention of the grant in the European Patent Bulletin (Art. 97(4), 99(1) EPC) November 13, 2002

Title of the invention: Detergent composition

II. Proprietor of the Patent

first named in the patent specification Quest International BV

Opponent's or representative's reference (max. 15 spaces) EP 1 149 149 OREF

III. Opponent

OPPO (2)

Name: The Procter & Gamble Company

Address: One Procter & Gamble Plaza
45202 Cincinnati, Ohio
U.S.A.

Zur Kasse
€610,-(A)

State of residence or of principle place of business: U.S.A.

Telephone/Telex/Fax: +1 513 945 9244 | +1 513 945 0000

Multiple opponents:

IV. Authorisation

1. Representative (Name only one representative to whom notification is to be made) OPPO (9)

Name: Jakob Kellenberger

Address of place of business: NV Procter & Gamble Services Cy SA
Temselaan 100
1853 Strombeek-Bever
Belgium

Telephone/Telex/Fax: + 32 2 456 3265 | + 32 2 456 3275

Additional representative(s): X (on additional sheet/see authorisation) OPPO (5)

2. Employee(s) of the opponent authorised for these opposition proceedings under act. 133(3) EPC

Name(s):

Authorisation(s)

To. 1./2.

not considered necessary

X has/have been registered under No. 2048

is/are enclosed

EPA Form 2300.1 04.93

for EPO use only

V. Opposition is filed against

— the patent as a whole [X]

— claim(s) No(s).

VI. Grounds for opposition:

Opposition is based on the following grounds:

(a) the subject-matter of the European patent opposed is not patentable (Art. 100(a) EPC) because

— it is not new (Art. 52(1); 54 EPC) [X]

— it does not involve an inventive step (Art.52(1); 56 EPC) [X]

— patentability is excluded on other grounds, i.e. Art. [ ]

(b) the patent opposed does not disclose the invention in a manner sufficiently clear and complete for it to be carried out by a person skilled in the art (Art. 100(b) EPC; see Art. 83 EPC). [X]

(c) the subject-matter of the patent opposed extends beyond the content of the application/ of the earlier application as filed (Art. 100(c)EPC, see Art. 123(2) EPC). [X]

VII. Facts and arguments

(Regel 55(c) EPC)
presented in support of the opposition are submitted herewith on a separate sheet (annex 1) X

VIII. Other requests:

Oral Proceedings are requested in the event that the patent opposed is not to be revoked as requested in the written submissions

EPA Form 2300.2 04.93

IX. Evidence presented

for EPO use only

Enclosed = X

will be filed at a later date =

A. Publications:	Publication date
1 GB 1 461 775 (D1) Particular relevance (page, column, line, fig.): Please refer to the statement of grounds	
2 US 5 126 061 (D2) Particular relevance (page, column, line, fig.): Please refer to the statement of grounds	
3 WO97/14780 (D3) Particular relevance (page, column, line, fig.): Please refer to the statement of grounds	
4 EP 0 397 246 (D4) Particular relevance (page, column, line, fig.): Please refer to the statement of grounds	
5 WO00/36078 (D5) Particular relevance (page, column, line, fig.): Please refer to the statement of grounds	
6 K.P. Guiseley, Enzyme Microb. Technol., (1989), Vol. 11, 706-715 (D6) Particular relevance (page, column, line, fig.): Please refer to the statement of grounds	
7 Particular relevance (page, column, line, fig.): Please refer to the statement of grounds	
Continued on additional sheet ☐	

B. Other evidence

Continued on additional sheet ☐

for EPO use only

X. Payment of the opposition fee is made

- [X] as indicated in the enclosed voucher for payment of fees and costs (EPO Form 1010)
- [ ]

XI. List of documents:

Enclosure No.			No. of copies	
0	[X]	Form for notice of opposition	2 (min. 2)	
1	[X]	facts and arguments (see VII.)	2 (min. 2)	
2		Copies of documents presented as evidence (see IX.)		
2a	[X]	— Publications	2 (min. 2 of each)	
2b	[ ]	— Other documents	(min. 2 of each)	
3	[ ]	Signed authorisation(s) (see IV.)		
4	[X]	Voucher of payment of fees and costs (see X.)	1	
5	[ ]	Cheque		
6	[X]	Additional sheet(s)	2 (min. 2 of each)	
7	[ ]	Other (please specify here):		

XII. Signature of opponent or representative

Place Strombeek-Bever

Date August 8, 2003

Jakob Kellenberger- Auth. No. 2048

Please type name under signature. In the case of legal persons, the position which the person signing holds within the company should also be typed.

EPO Form 2300.4 04.93

Procter&Gamble

N.V. Procter & Gamble Services Company S.A.
Temselaan 100, B-1853 Strombeek-Bever, België

European Patent Office
Directorate General 2
Erhardtstrasse 27
D-80298 Munich

GERMANY

DHL

August 8, 2003

NOTICE OF OPPOSITION

Opposition filed by :
The Procter & Gamble Company
One Procter & Gamble Plaza
45202 Cincinnati, Ohio
U.S.A.

Against: EP 1 149 149 B, granted on November 13, 2002
Proprietor: Quest International BV

Grounds for Opposition

Statement of facts and arguments of Opposition to European Patent No. 1 149 149 B, granted to QUEST INTERNATIONAL B.V., on November 13, 2002, and corresponding to European Patent Application No. 00901233.7, claiming priority of two earlier GB Patent applications filed February 2, 1999, and March 16, 1999.

Opposition submitted by The Procter & Gamble Company.

1. EP-B-1 149 149

1.1 EP-B-1 149 149 (hereinafter referred to as the Patent) was granted with 13 claims, consisting of three independent claims (claims 1, 10 and 11) and 10 dependent claims (claims 2-9, 12 and 13).

1.2 Independent claim 1 of the Patent relates to a liquid detergent composition comprising at least 5% by weight of surfactant and an encapsulate comprising greater than 10% by weight of the encapsulate of active material, in a hydrated cross-linked anionic gum matrix.

1.3 Independent claim 10 of the Patent relates to a liquid detergent composition comprising at least 5% by weight of surfactant and an encapsulate comprising greater than 0.5% by weight of the encapsulate of fragrance, in a hydrated cross-linked anionic gum matrix.

./..

☎ +32-(0)2 456 21 11 - TELEFAX: +32-(0)2 456 45 70 - CABLE: Centerproc Bru
BBL Brussel / Bruxelles: 310-1147902-20 - B.T.W./T.V.A.: BE 437.862.552 - H.R.B./R.C.B.: 521.494

1.4 Independent claim 11 of the Patent relates to a laundry liquid detergent composition comprising at least 5% by weight of surfactant and an encapsulate comprising active material in a hydrated cross-linked anionic gum matrix.

1.5 Dependent claims 2-9, 12 and 13 relate to specific embodiments of the alleged invention.

2. Prior Art

2.1 The Opponent will show that the Patent lacks novelty and inventive step in view of documents D1 to D6 as listed below :

D1 :	GB 1 461 775	UNILEVER Ltd.
D2 :	US 5,126,061	The Procter & Gamble Company
D3 :	WO 97/14780	UNILEVER N.V. / PLC
D4 :	EP-A-0 397 246	The Procter & Gamble Company / 3M
D5 :	WO 00/36078	UNILEVER N.V. / PLC
D6 :	K. P. Guiseley, Enzyme Microb. Technol., (1989), Vol. 11, 706-715	

3. Lack of Novelty

3.1 The alleged Invention as claimed by the Patent lacks novelty in view of D1 to D5 as described in more detail below.

3.2 Lack of Novelty in view of D1 (GB 1 461 775)

3.2.1 D1 filed on April 27, 1973, published January 19, 1977, relates to aqueous detergent compositions.

3.2.2 D1 describes liquid aqueous detergent compositions comprising suspended particles containing a beneficial agent (D1, page 1, lines 10-14).

3.2.3 D1 exemplifies liquid detergent compositions (D1, pages 2+3, EXAMPLES 1 (a)-(e)) comprising water and 17.2% of triethanolamine lauryl sulphate (Empicol TL 40; 43% of a 40% active solution) as anionic surfactant. Furthermore, EXAMPLES 1 (a)-(e) additionally comprise encapsulates (particles / capsules) comprising 15% by weight of the encapsulate of a water-dispersible pigment (Monastral Blu BV) as active material in a hardened (D1, page 1, line 40) ι-carrageenan and/or κ-carrageenan gum matrix (D1, page 3, lines 10-38; (ii) particles).

3.2.4 The Opponent respectfully submits that the Patent in section [0019] specifically mentions colouring materials as suitable actives for use in the alleged invention. Therefore, the water-dispersible pigment of EXAMPLES 1 (a)-(e) of D1, is an active material as required by the Patent. Moreover, perfumes are listed in D1 as additional ingredients for use in the encapsulates described therein (D1, page 2, lines 95-99).

./..

3.2.5 In addition, it is submitted that carrageenan gums are listed in section [0026] of the Patent as suitable anionic gums for use therein and in claim 8 as preferred anionic gums. Furthermore, it is submitted that the carrageenan gums of the particles described in EXAMPLES 1 (a)-(e) are hydrated as they are in a water-containing composition.

3.2.6 Moreover, the carrageenan gums of the particles described in EXAMPLES 1 (a)-(e) are crosslinked by adding the droplets formed into a 2.5% potassium chloride hardening bath (D1, page 3, lines 35-38). Indeed, sections [0026] and [0037], lines 40+41, of the Patent describe the cross-linking of carrageenan gums using potassium ions. Furthermore, D6, page 709, col. 2, lines 5-11, provides further evidence of the gelling / cross-linking behavior of carrageenan gums in the presence of potassium ions. It is submitted that the terms : hardening, cross-linking and gelling; describe the same technical effect and will hence be regarded as synonymous for the purpose of the present opposition.

3.2.7 In addition, EXAMPLES 1 (a)-(e) describe encapsulates wherein the ι-carrageenan and/or κ-carrageenan anionic gum is present in an amount between 2% and 4% by weight of the encapsulate.

3.2.8 The Opponent respectfully submits that the term "laundry" in independent claim 11 of the Patent is not considered as limiting the scope of this claim.

3.2.9 Hence, in view of the above, independent claims 1 and 11 as well as dependent claims 2-8 and 12 of the Patent lack novelty over D1.

3.3 Lack of Novelty in view of D2 (US 5,126,061)

3.3.1 D2 filed July 18, 1990, and granted on June 30, 1992, relates to microcapsules containing a hydrophobic liquid core (perfume / fragrance).

3.3.2 D2 exemplifies a liquid composition (D2, cols. 17+18, TABLE 2, Composition A) comprising water and 14.18% of a surfactant mixture (7.97% Adogen® 44E-83HM and 6.21% Variosoft® 445). Furthermore, Composition A additionally comprises 0.9% of encapsulates (Perfume capsules) of type 1 (D2, col. 17, line 68, - col. 18, line 2).

3.3.3 The perfume capsule of type 1 is described in TABLE 1 of D2 as Microcapsule 1. Microcapsule 1 comprises 15% by weight of the encapsulate including water (see the Patent, section [0020], last sentence) of perfume / fragrance (Total perfume in Microcapsule 1 is 125 g in a total of 800 g) as active material in a crosslinked (D2, col. 17, 35-37, and TABLE 1, last two lines : Cross-linking time) gelatin- gum arabic gum matrix.

3.3.4 It is submitted that gum arabic is listed in section [0029] of the Patent as a suitable anionic gum for use therein. Furthermore, it is submitted that the gelatin- gum arabic gum matrix of the Perfume capsules described in Composition A are hydrated as they are in a water-containing composition.

3.3.5 In addition, Microcapsules 1 of TABLE 1 comprise 1.25% by weight of the encapsulate including water of gum arabic (10 g gum arabic in a total of 800 g).

./..

3.3.6 The Opponent submits that Compositions B-G in TABLE 2 of D2 exemplify similar liquid compositions.

3.3.7 Hence, in view of the above, independent claims 1, 10 and 11 as well as dependent claims 2, 3 and 5-7 of the Patent lack novelty over D2.

3.4 Lack of Novelty in view of D3 (WO 97/14780)

3.4.1 D3 filed on September 19, 1996, with a priority date of October 16, 1995, published April 24, 1997, relates to encapsulated bleach particles.

3.4.2 D3 describes encapsulated bleach particles (D3, claim 1) comprising 1-30% by weight (of the encapsulate) of a coating including gelled polymer material. The gelled polymer material is selected from the group of alginate, carrageenan and gellan gum (D3, page 3, lines 25-29), with alginate as the preferred polymer material (D3, page 3, line 29, and claim 2). It is submitted that alginate, carrageenan and gellan gum are listed in section [0026] and claim 8 of the Patent as a suitable/ preferred anionic gum for use therein. In addition, the gelled polymer material is partially or fully crosslinked (D3, page 3, lines 20-24).

3.4.3 Furthermore, D3 describes encapsulated bleach particles (D3, claim 1) comprising 99-70% by weight (of the encapsulate) of a core material selected from the group consisting of a peroxygen bleach compound, a bleach catalyst, and a peroxygen bleach precursor, as active material.

3.4.4 Moreover, D3 describes that the encapsulated bleach particles are incorporated into a bleaching detergent, which is in liquid form (D3, page 14, lines 19-21) and preferably comprises from 1-15% of anionic surfactant and from 10-40% of nonionic surfactant (D3, page 11, last paragraph) as well as from 5-80% of a builder (D3, page 12, lines 1-5). The Opponent respectfully submits that the anionic gum matrix of the encapsulated bleach particles described in D3 is hydrated once introduced into a liquid composition.

3.4.5 Hence, in view of the above, independent claims 1 and 11 as well as dependent claims 2-4, 7-9 and 13 of the Patent lack novelty over D3.

3.5 Lack of Novelty in view of D4 (EP-A-0 397 246)

3.5.1 D4 filed on May 2, 1990, with a priority date of May 11, 1989, published November 14, 1990, relates to coated perfume particles.

3.5.2 D4 exemplifies a liquid laundry detergent composition (D4, pages 9+10, Example VI) comprising water and 25.7% of a surfactant mixture (7.2% C_{13} linear alkylbenzene sulphonic acid and 10.8% C_{14-15} alkyl polyethoxylate (2.25) sulfuric acid as anionic surfactants, 6.5% C_{12-13} alcohol polyethoxylate (6.5) as nonionic surfactant and 1.2% C_{12} alkyl trimethylammonium chloride as cationic surfactant). Furthermore, the liquid laundry detergent composition of Example VI comprises 1% of encapsulates (perfume-containing particles; D4, page 10, lines 37-41) as prepared in Example II of D4.

./..

3.5.3 The perfume-containing particles are prepared in Example II of D4 (same amounts used as in Example I of D4) in a solution comprising 5.612% by weight of the solution of perfume / fragrance (perfume in the solution of Example II is 44 g in a total of 784 g) as active material in a gluteraldehyde crosslinked (D4, page 7, lines 12-15 and lines 47-49) gelatin- gum arabic gum matrix.

3.5.4 However, from Example VI of D4 it becomes apparent that the perfume particles prepared in Example II have a final level of active, i.e., perfume, of 33%. Indeed, from the statement on page 10, lines 40+41, : ... the detergent composition comprises about 0.3% perfume (about 1% of the detergent composition will comprise the perfume particles).; it can be concluded that the final level of perfume (active material) in the particle is 33%. The Opponent submits that even though the perfume particles as described in Example II are formed using solution comprising 5.612% perfume, the formation of the perfume particles is performed in an excess of water. In conclusion, the fully hydrated perfume particles of Example II of D4 show an active material level of 33%.

3.5.5 It is submitted that gum arabic is listed in section [0029] of the Patent as a suitable anionic gum for use therein. Furthermore, it is submitted that the gelatin- gum arabic gum matrix of the Perfume capsules described in Example VI is hydrated as it is in a water-containing composition.

3.5.6 In addition, the solution of Example II comprise 2.806% by weight of gum arabic (200 g of an 11%-active gum arabic solution in a total of 784 g).

3.5.7 Hence, in view of the above, independent claims 1, 10 and 11 as well as dependent claim 2-7 and 12 of the Patent lack novelty over D4.

3.6 Lack of Novelty in view of D5 (WO 00/36078)

3.6.1 D5 filed on November 11, 1999, with a priority date of December 16, 1998, published June 22, 2000, relates to pourable transparent / translucent liquid detergent compositions with suspended particles.

3.6.2 D5 is prior art under Art. 54(3) EPC for AT, BE, CH, CY, DE, DK, ES, FI, FR, GB, GR, IE, IT, LI, LU, MC, NL, PT and SE, which are designated in common in D5 and the Patent.

3.6.3 D5 exemplifies a liquid detergent composition (D5, pages 41+42, Example 10) comprising water and 27.75% of a surfactant mixture (20.0% of alcohol ethoxylate as nonionic surfactant and 7.75 of sodium linear alkyl benzene sulfonate as anionic surfactant). Furthermore, Example 10 comprises 1% of encapsulates (capsules) as prepared in Example 1 of D5.

3.6.4 The capsules in Example 1 (D5, pages 39+40) of D5 comprise 3% by weight of the encapsulate including water of Zeolite (90 g of Zeolite in a total of 3000 g) and 1% by weight of the encapsulate including water of white pigment (30 g of white pigment in a total of 3000 g) as active materials.

./..

3.6.5 The Opponent respectfully submits that the Patent in section [0019] specifically mentions colouring materials as suitable actives for use in the alleged invention. Therefore, the white pigment of Example 1 of D5 is an active material as required by the Patent. Furthermore, Zeolite, a detergent builder (D5, page 34, 3rd paragraph) is also an active material as required by the Patent.

3.6.6 The capsules in Example 1 comprise 2% by weight of the encapsulate including water of a hardened κ-carrageenan gum matrix (D5, page 40, 1st paragraph). It is submitted that carrageenan gum is listed in section [0026] of the Patent as a suitable anionic gum for use therein and in claim 8 as a preferred anionic gum. Furthermore, it is submitted that the carrageenan gum of the capsules described in Example 10 are hydrated as they are in a water-containing composition.

3.6.7 The κ-carrageenan gum of the particles described in Example 1 is crosslinked by spraying the composition into a 5% potassium chloride hardening bath (D5, page 40, 1st paragraph). Indeed, sections [0026] and [0037], lines 40+41, of the Patent describe the cross-linking of carrageenan gum using potassium ions. Furthermore, D6, page 709, col. 2, lines 5-11, provides further evidence of the gelling / cross-linking behavior of carrageenan gums in the presence of potassium ions.

3.6.8 The Opponent submits that Examples 6-9, 11 and 19-21 on pages 41-43 and 50+51 of D5 exemplify similar liquid compositions as described above. Furthermore, Examples 12-18 on pages 43-50 of D5 exemplify compositions comprising encapsulates as prepared in Example 5 (D5, pages 40+41) with pigment as active material and gellan gum (Kelcogel LT) forming a crosslinked anionic gum matrix.

3.6.9 Furthermore, the liquid composition of Example 17 additionally comprises 5.6% of sodium silicate (44.46 g of sodium silicate in a total of 793.46 g) as builder (D5, page 22, 4th paragraph).

3.6.10 Hence, in view of the above, independent claim 11 as well as dependent claims 12+13 of the Patent lack novelty over D5.

4. Lack of Inventive Step

4.1 The problem addressed by the Patent is that of providing a dilution release encapsulate, which can be incorporated in a laundry liquid base, is stable on storage yet dissolves on dilution of the product, thereby releasing the active material (section [0018]).

4.2 The solution provided by the Patent is a liquid detergent composition comprising at least 5% by weight of surfactant and an encapsulate comprising active material in a hydrated cross-linked anionic gum matrix (claim 11).

4.3 Lack of Inventive Step in view of D1 (GB 1 461 775)

4.3.1 The Opponent submits that independent claims 1 and 11 as well as dependent claims 2-8 and 12 of the Patent (see paragraphs 3.2.2 to 3.2.7) are fully anticipated by D1. Should the Opposition Division not follow the Opponents arguments regarding novelty

./..

4.3.2 of independent claims 1 and 11 as well as dependent claims 2-8 and 12 of the Patent in view of D1, the Opponent respectfully submits that independent claims 1 and 11 as well as dependent claims 2-8 and 12 of the Patent lack inventive step over D1 in view of the arguments brought forward in paragraphs 3.2.2 to 3.2.7.

4.3.3 Independent claim 10 of the Patent differs from D1 only in that it requires the presence of greater than 0.5% by weight of the encapsulate of fragrance in the encapsulates therein. Indeed, the Opponent respectfully submits that D1 describes encapsulates that can additionally comprise perfumes (D1, page 2, lines 95-99).

4.3.4 The Patent is completely silent about the benefit provided by the specific level of greater than 0.5% by weight of the encapsulate of fragrance. It is thus submitted that the choice of level of fragrance in the encapsulate relates to a mere alternative of the Invention, if any, according to the Patent over the prior art. Indeed, no surprising effect is connected to the choice of level of fragrance in the encapsulate and hence it relates to the obvious application of a measure resulting in no benefit, which cannot be regarded as inventive. Furthermore, the choice of fragrance / perfume as active material in the encapsulates cannot be regarded as inventive in view of the statement in D1 that the encapsulates described therein can additionally comprise perfumes (D1, page 2, lines 95-99).

4.3.5 Claim 8 dependent from claim 1 of the Patent differs from D1 only in that it requires that the anionic gum is alginate, whereas the anionic gums in D1 are carrageenan gums. From section [0027] and the last sentence in section [0029], it can be concluded that alginates are preferred anionic gum in the Patent for their improved stability. Hence, the problem of providing encapsulates having an improved stability constitutes the objective problem of claim 8 of the Patent in view of D1.

4.3.6 D6 is a review discussing the properties of different algal polysaccharides, such as alginates and carrageenan gums. One teaching of D6 is that gels formed by crosslinking alginates are more stable as compared to gels formed by crosslinking carrageenan gums due to the fact that alginate gels are thermally irreversible gels, whereas carrageenan gums gels are thermally reversible (D6, page 708, col. 2, lines 9-12 and 55+56, and page 709, col. 2, lines 30-32).

4.3.7 The Opponent submits that a person skilled in the art reading D1 and faced with the objective problem as described above, would consider document D6 when trying to increase the stability of the encapsulates formed in D1. Indeed, D6 is a general review of the properties of algal polysaccharides and constitutes general common knowledge with regard to the properties of anionic gums. The disclosure of D6 teaches the skilled person that the choice of alginates as the anionic gum to form encapsulates will yield in encapsulates having an improved stability.

4.3.8 In view of the above it is submitted that it would have been obvious for the skilled person faced with the above objective problem and when reading D1 in combination with D6, to choose alginates as the anionic gum to form encapsulates when faced with the problem of providing encapsulates having improved stability.

./..

4.3.9 In view of the arguments brought forward in paragraphs 4.3.1 to 4.3.7, the Opponent submits that independent claim 10 lacks inventive step in view of D1 and that dependent claim 8 lacks inventive step in view of D1 in combination with D6.

4.4 Lack of Inventive Step in view of D2 (US 5,126,061)

4.4.1 The Opponent submits that independent claims 1, 10 and 11 as well as dependent claims 2, 3 and 5-7 of the Patent (see paragraphs 3.3.2 to 3.3.6) are fully anticipated by D2. Should the Opposition Division not follow the Opponents arguments regarding novelty of in independent claims 1, 10 and 11 as well as dependent claims 2, 3 and 5-7 of the Patent in view of D2, the Opponent respectfully submits that independent claims 1, 10 and 11 as well as dependent claims 2, 3 and 5-7 of the Patent lack inventive step over D2 in view of the arguments brought forward in paragraphs 3.3.2 to 3.3.6.

4.4.2 The novel features in view of D2 of dependent claims 4, 8, 9, 12 and 13 of the Patent relate to the application and variation of conventional features that are obvious for the skilled person. Hence, claims 4, 8, 9, 12 and 13 of the Patent cannot be regarded as involving an inventive step over D2.

4.5 Lack of Inventive Step in view of D3 (WO 97/14780)

4.5.1 The Opponent submits that independent claims 1 and 11 as well as dependent claims 2-4, 7-9 and 13 of the Patent (see paragraphs 3.4.2 to 3.4.4) are fully anticipated by D3. Should the Opposition Division not follow the Opponents arguments regarding novelty of in independent claims 1 and 11 as well as dependent claims 2-4, 7-9 and 13 of the Patent in view of D3, the Opponent respectfully submits that independent claims 1 and 11 as well as dependent claims 2-4, 7-9 and 13 of the Patent lack inventive step over D3 in view of the arguments brought forward in paragraphs 3.4.2 to 3.4.4.

4.5.2 Independent claim 10 of the Patent differs from D3 only in that it requires the presence of greater than 0.5% by weight of the encapsulate of fragrance in the encapsulates therein. Furthermore, dependent claim 5 of the Patent differs from D3 only in that it requires the presence of a fragrance in the encapsulates therein.

4.5.3 The Patent fails to provide a specific benefit for the choice of fragrance as active material as compared to other active materials, as described in section [0019] of the Patent. Moreover, the Patent is completely silent about the benefit provided by the specific level of greater than 0.5% by weight of the encapsulate of fragrance. It is thus submitted that the choice of fragrance as the active material and the level of fragrance in the encapsulate relate to mere alternatives of the Invention, if any, according to the Patent over the prior art. Indeed, no surprising effect, other than the obvious perfume benefit provided by a fragrance, is connected to the choice active material or to the choice of level of fragrance in the encapsulate and hence it relates to the obvious application of known measures resulting in no benefit, which cannot be regarded as inventive.

./..

4.5.4 In view of the arguments brought forward in paragraphs 4.5.1 to 4.5.3, the Opponent submits that independent claim 10 and dependent claim 5 of the Patent lack inventive step in view of D3.

4.6 Lack of Inventive Step in view of D4 (EP-A-0 397 246)

4.6.1 The Opponent submits that independent claims 1, 10 and 11 as well as dependent claim 2-7 and 12 of the Patent (see paragraphs 3.5.2 to 3.5.6) are fully anticipated by D4. Should the Opposition Division not follow the Opponents arguments regarding novelty of in independent claims 1, 10 and 11 as well as dependent claim 2-7 and 12 of the Patent in view of D4, the Opponent respectfully submits that independent claims 1, 10 and 11 as well as dependent claim 2-7 and 12 of the Patent lack inventive step over D4 in view of the arguments brought forward in paragraphs 3.5.2 to 3.5.6.

4.6.2 The novel features in view of D4 of dependent claims 8 and 9 of the Patent relate to the application and variation of conventional features that are obvious for the skilled person. Hence, claims 8 and 9 of the Patent cannot be regarded as involving an inventive step over D4.

Yours faithfully,

Jakob Kellenberger
(Auth No 2048)

JK/vdk

BROOKS, Maxim, Courtney
Procter & Gamble Technical Centres Ltd.,
Whitley Road,
Longbenton
Newcastle upon Tyne NE12 9TS, Great Britain

CANONICI, Jean-Jacques
NV Procter & Gamble Services Company SA
Temselaan 100
1853 Strombeek-Bever, Belgium

CLEMO, Nicholas Graham
Procter & Gamble Technical Centres Ltd.,
Rusham Park, Whitehall Lane,
Egham, Surrey TW20 9NW
Great Britain

ENGISCH, Gautier
NV Procter & Gamble Services Company SA
Temselaan 100
1853 Strombeek-Bever, Belgium

GOODIER, Claire-Louise
NV Procter & Gamble Services Company SA
Temselaan 100
1853 Strombeek-Bever, Belgium

HIRSCH, Uwe Thomas
Procter & Gamble Service GmbH
Sulzbacher Strasse 40 – 50
65824 Schwalbach am Taunus, Germany

KOHOL, Sonia
Procter & Gamble Technical Centres Ltd.,
Rusham Park, Whitehall Lane,
Egham, Surrey TW20 9NW
Great Britain

KREMER,Véronique Marie Josephine
Procter & Gamble Service GmbH
Sulzbacher Strasse 40 - 50
65824 Schwalbach am Taunus, Germany

MORELLE, Evelyne
NV Procter & Gamble Services Company SA
Temselaan 100
1853 Strombeek-Bever, Belgium

PEET, Jillian Wendy
Procter & Gamble Technical Centres Ltd.,
Whitley Road,
Longbenton
Newcastle upon Tyne NE12 9TS, Great Britain

VERONESE, Pancrazio
Procter & Gamble Italia S.p.A. Italian Research Center
Via Aterno 92/94
66020 Sambuceto di San.Giovanni Teatino (Chieti)
Italy

WILDING, Richard Alan
Procter & Gamble Technical Centres Ltd.,
Rusham Park, Whitehall Lane,
Egham, Surrey TW20 9NW
Great Britain

BORBACH, Markus H. M
Procter & Gamble Service GmbH
Sulzbacher Strasse 40 - 50
65824 Schwalbach am Taunus, Germany

MCGREGOR Judith Ester
Procter & Gamble International Operations SA
Legal and Patent Department
47, Route de Saint Georges
CH-1213 Pet Lancy, Switzerland

MATHER, Peter Geoffrey
NV Procter & Gamble Services Company SA
Temselaan 100
1853 Strombeek-Bever, Belgium

Jakob KELLENBERGER
(Auth. No 2048)

KEITH W NASH & Co.

Patent and Trade Mark Attorneys

Partners: **K. W. Nash** B.Sc. C.P.A. E.P.A.
H. C. Matthews M.A. (Cantab) C.P.A. E.P.A.
D. L. Roberts B.Sc. C.P.A. E.P.A.
M. J. Lipscombe Ph.D. C.P.A. E.P.A.
H. J. Forsyth Ph.D. C.P.A. E.P.A.

Cambridge: **90-92 Regent Street**
Cambridge CB2 1DP
Tel: 01223 355477
Fax: 01223 324353
mail@kwnash.co.uk

Norwich: 72 Thorpe Road
Norwich NR1 1BA
Tel: 01603 614832
Fax: 01603 616847
mail@kwnash.co.uk

Associates: **A. D. Branscombe** B. Eng.
C. A. Roberts B.Sc

Consultants: **C. D. Morton** M.A. (Cantab) C.P.A. E.P.A.
C. D. Benziger H.N.D. C.P.A. E.P.A.
M. Waggett Dipl. El. C.P.A. E.P.A.

Please correspond with Cambridge Office

Our Ref : HCM/CAR/SS/C281.00/Q
Your Ref :
Date : 30th April 2004

COPY

European Patent Office
D-80298 Munich
GERMANY

Dear Sirs

re: European Patent Application No. 00901233.7 (Patent No. 1149149)
Quest International B.V.
Opposition thereto by The Procter & Gamble Company

We refer to the Communication of a Notice of Opposition dated 17th September 2003 concerning the opposition filed by The Procter & Gamble Company (the Opponent).

In response, we file herewith a set of amended claims for consideration. We believe the amended claims are patentable over the prior art, and request that the Opposition Division maintain the patent on the basis of the amended claims. The amendments do not constitute the abandonment of subject matter, and we reserve the right to revert to the claims as granted and/or to file further amended claims at a later stage.

In the amended claims, claim 1 has been amended to require that the encapsulate of the liquid detergent composition comprises greater than 10% by weight of the encapsulate of active material comprising a fragrance in a hydrated cross-linked anionic gum matrix, based on claim 5 as granted. Claim 5 has been deleted and the remaining claims as granted renumbered accordingly.

New claim 10 (claim 11 as granted) has also been amended, and requires that the encapsulate of the laundry liquid, comprises fragrance in a hydrated cross-linked anionic gum matrix. This amendment is based on paragraph No. 0019 of the patent specification, line 35.

New claim 13 has been added based on claim 1 as granted and paragraph Nos. 0018, lines 27-29, and 0019, lines 30-32.

New claim 14 is based on claim 11 as granted and paragraph Nos. 0018, lines 27-29 and 0019, lines 30-32.

We note that the Opponent has indicated in EPA Form 2300 that the patent is opposed on the grounds that the patent does not disclose the invention in a manner sufficiently clear and complete for it to be carried out by a person skilled in the art (Art. 100(b) EPC), and the subject matter of the patent extends beyond the content of the application/of the earlier application as filed (Art. 100(c) EPC). However, neither of these grounds have been substantiated by the Opponent in the facts and

arguments presented, so these grounds will not be considered further and we will confine our reply to discussing the prior art relied on.

As discussed in the 'Background to the Invention' of the patent (see paragraph No. 0002), encapsulate systems fall into 2 main types: those in which the active material (fragrance/enzyme) is surrounded by a wall or barrier, and those in which the active material (fragrance/enzyme) is encapsulated in the matrix of a material.

In this connection, we enclose a copy of an article entitled "Encapsulation: New Tools for Ingredient Delivery" by Hetherington (A1), which gives a very useful description of how these different encapsulate forms may be visualised. Paragraph 5 of this article states that core/shell capsules can be thought of as an M&M candy where the core material is the peanut and the shell is the chocolate coating. Matrix encapsulates, on the other hand, can be thought of as a sponge with the active entrapped in the voids inside the sponge, so that some of the active can be seen from the outside.

Different types of encapsulation are also described in Jacobs, I.C. and N.S. Mason, Polymeric delivery systems, properties and applications, Edited by M.A.El-Nokaly, D.M. Piatt, and B.A. Charpentier, American Chemical Society Symposium Series 520, ISBN 0-842-2624-5, pages 3-4 (1993) (A2). This includes reservoir devices or encapsulates and matrix types. Specifically, reservoir devices consist of a drug or other active agent enclosed within an inert controlling membrane. Examples would be a tube filled with the active substance, where the wall of the tube would serve as the limiting membrane, a sphere of the active substance coated with a film controlling the diffusion of the active substance, or a slab of the active substance closed off from the medium by a film which controls the diffusion. Matrix devices, on the other hand, have the active material dispersed throughout the polymer and are called monolithic devices or monoliths. One can distinguish between monolithic devices in which the active ingredient is dissolved, dispersed, located in connected pores, or granular.

The encapsulates useful in the liquid detergent compositions of independent claims 1, 9 and 13 and the laundry liquids of independent claims 10 and 14 are matrix encapsulates, and more particularly encapsulates comprising fragrance or enzyme in a hydrated cross-linked anionic gum matrix.

Against this background, the Opponent has cited US 5,126,061 (D2) and EP 0397246 (D4) as full prior art against the claims of the patent.

US 5,126,061 (D2) generally concerns microcapsules containing a central core of hydrophobic liquid, where the microcapsules preferably have complex wall structures in which the capsule walls surrounding the central core comprise substantial amounts of relatively small wall inclusion particles which can be activated by heat to disrupt the wall.

The microcapsules of D2 are prepared by the process of coacervation, e.g. from gelatin and a polyanionic material, and cross-linked with glutaraldehyde. In this process, a soluble mixture of gelatin and arabic gum are brought out of solution to form a shell wall around droplets of core material, e.g. perfume. This shell wall is then hardened, e.g. by addition of glutaraldehyde. The process results in the formation of a microcapsule having a discrete wall around emulsified hydrophobic liquid droplets. According to column 2, lines 32-38 of the patent specification of D2, the microcapsules may be visualised as shown in Figures 1 and 2 of US 3,888,689 (A3, copy attached). Figure 1, in particular, clearly shows the wall structure of the microcapsules.

Thus, there is no disclosure in D2 of encapsulates having a matrix structure as required by the present claims, let alone a structure of hydrated cross-linked anionic gum matrix. As noted above, D2 merely describes encapsulates having a wall structure.

D2 is therefore of no relevance to the presently claimed invention, and we propose not to discuss the document further.

EP 0397246 (D4) concerns perfume particles comprising perfume dispersed in a water-insoluble polymeric carrier material of specified properties, the particles having a substantially water-insoluble friable coating on their outer surfaces. The perfumed particles are coated with a friable coating material which ruptures in-use to release the perfume particle which, in turn, releases perfume (see page 4, lines 26-27). The document at page 6, lines 49-56, discloses that the coating is applied to the perfumed particles as a kind of "shell" to encapsulate the solid particles. Examples of suitable encapsulating materials and processes are disclosed at page 7, lines 9-12 as including gelatin-gum arabic concentrates deposited by a complex coacervation procedure or ureaformaldehyde deposited by a polycondensation process.

The perfume particles of D4 are therefore wall encapsulates rather than matrix encapsulates of the present invention. Therefore, there is no disclosure in D4 of a liquid detergent composition or laundry liquid comprising at least 5% by weight of surfactant and or an encapsulate comprising fragrance/enzyme in a hydrated cross-linked anionic gum matrix.

For the above reasons, it is clear that D4 is also of no relevance to the presently claimed invention and similarly the document can be disregarded.

GB 1461775 (D1) is available as full prior art.

This document concerns liquid aqueous detergent compositions comprising a clear liquid phase having suspending properties, comprising from 1-45% by weight of detergent active organic material and having dispersed and suspended therein particles, e.g. of hardened carrageenan and a water-dispersible pigment (page 1, column 1, lines 33-43).

There is a passing reference at page 2, column 2, lines 90-99, that the liquid phase or the particles can additionally comprise, amongst many other ingredients, perfumes. However, the examples of the document use particles containing liquid paraffin and water-dispersible pigment. There is no mention of enzymes as possible ingredients. In particular, Example 1 discloses bath additives containing particles. The bath additives contain 17.2% w/w of the liquid phase of triethanolamine lauryl sulphate and particles (a)-(e) (see page 3, column 1, lines 10-30). The particles containing liquid paraffin are prepared by the concentric orifice technique using 1-4% w/w aqueous solutions of carrageenan as wall materials, mixing each of the solutions with 15% w/w of Monastral Blue BV as the water-dispersible pigment, and hardening the droplets containing oil by dropping them into a bath containing 2.5% w/w potassium chloride in a 3:1 mixture by weight of ethanol and water. Example 2 also describes a bath additive containing 17.2% w/w of the liquid phase of triethanolamine lauryl sulphate, and particles containing liquid paraffin prepared by the concentric orifice technique from a dispersion of wall material including 3% w/w aqueous solution of carrageenan (70% w/w), Monastral Blue BV (18% w/w) and other pigments. The droplets containing oil are hardened by dropping them into a bath containing 2.5% w/w potassium chloride in a 3:1 mixture by weight of ethanol and water.

The finished particles of D1 not only do not contain perfume or enzyme, but are wall encapsulates. The concentric orifice technique used to prepare the particles is a method employed for forming wall

encapsulates. The apparatus consists of two nozzles, one nestled inside the other, with the inner nozzle containing core material and the outer nozzle containing wall material.

D1 therefore, explicitly nor implicitly, discloses all of the features of the present claims, and, in particular, an encapsulate including fragrance or enzyme in a hydrated cross-linked anionic gum matrix. The presently claimed invention is therefore novel over D1.

The Opponent alleges at paragraph 3.2.8 of the Statement of Facts and Arguments that the term "laundry" in independent claim 11 of the patent as granted (new claim 10) is not considered as limiting the scope of the claim. We do not believe that this is an appropriate interpretation. The term "laundry liquid" has a specific meaning, which would be understood by a person of ordinary skill in the art. A laundry liquid is clearly a liquid product as sold: formulated for the sole purpose of laundry use. As discussed in the patent specification at paragraph Nos. 0041 to 0052, laundry liquids are products generally containing additives such as builders, e.g. for increasing the efficiency and effectiveness of surfactants and to supplement their beneficial effects on soil removal; soil release agents; anti-redeposition agents; fillers; fluorescent brighteners; bleaches etc. Such products are therefore wholly inappropriate for topical use on the body, e.g. skin or hair, or as bath additives. For this reason alone, we submit that D1 is not relevant to claims 10 and 14.

WO 97/14780 (D3) is available as full prior art.

The document concerns an encapsulated bleach particle comprising a coating including gelled polymer material and a core material selected from a peroxy bleaching agent, a bleach catalyst and a peroxygen bleach precursor. The gelled polymer of the coating is partially or fully cross-linked, e.g. with alkaline earth metals (see page 3, lines 20-24 and page 3, line 34 to page 4, line 1). The polymer may be selected from a number of materials, e.g. alginate, carrageenan, gelatin; with alginate being preferred. The core material may constitute 70-99% by weight of the particle.

The process for producing the coated particles is described in detail at page 9, line 10 to page 10, line 3. Briefly, the process involves atomising an aqueous suspension containing both the polymer to be gelled and the core material; gelling the thus obtained droplets; and drying the gelled droplets to from dry-free flowing particles.

The document discloses that the particles may be added to bleaching detergent compositions comprising surfactant material and builder material. The bleaching detergent compositions are disclosed as generally containing 10-50% by weight of surfactant material (see page 10, lines 6-8), particularly 1-15% by weight of anionic surfactant material (see page 10, lines 6-8), particularly 1-15% by weight of anionic surfactant and 10-40% by weight of non-anionic surfactant, and 5-80% by weight of a detergency builder (see page 12, lines 1-5).

Perfumes and enzymes are mentioned in the document in the context of possible additives for bleaching detergent compositions, and not encapsulates.

There is a passing reference to the bleaching detergent composition being formulated in liquid form (see page 14, lines 19-21). However, the specific bleaching detergent compositions contemplated in D3 are dry products, see e.g. page 14, lines 22-37. Moreover, there are no explicit examples of liquid detergents in the document. The examples on pages 16-23 simply describe particulate detergent comprising the encapsulated bleach particles.

D3 therefore neither teaches nor suggest encapsulates comprising fragrance or enzyme in a hydrated cross-linked anionic gum matrix, let alone liquid detergent compositions or laundry liquids comprising such encapsulates.

The presently claimed invention is therefore novel in view of D3.

WO 00/36078 (D5) is citable under Article 54(3) EPC as of possible relevance to novelty only.

The document concerns heavy duty liquid laundry detergents comprising polymers, e.g. gums, capable of forming suspending networks in the presence of surfactants and/or electrolytes to suspend particles, e.g. capsules. The compositions comprise greater than 15% by weight of surfactant. Pages 33-34 of D5 disclose a number of possible ingredients that can be incorporated in capsules. Mention is made of perfumes and enzymes, amongst many other materials including fabric softening agents, anti-foams, builder zeolite, and fabric protection polymers and soil release polymers, e.g. polyvinylpyrrolidone (PVP).

Examples 1-5 on pages 39 to 41 of the document describe the preparation of a number of capsules, including zeolite (Examples 1, 3 and 4), white pigment (Example 1), fluorescent dye (Example 2) and PVP (Examples 2, 3 and 4). Example 1, in particular, describes capsules prepared from K-carrageenan gum (2%), water (94%), zeolite (3%) and white pigment (1%), and Example 5 describes capsules prepared from gellan gum (0.5%), water (97%), sodium citrate (0.1%) and pigment (0.97%). The capsules of Example 1 are added to exemplified liquid detergents 6-11 and 19-21. Capsules of Example 5 are added to exemplified liquid detergents 12-18. Of these, Example 17 discloses a liquid detergent which comprises 5.7% alcohol ethoxylate (surfactant); encapsulates of example 5; and 5.6% sodium silicate (i.e. builder).

We accept that D5 discloses a laundry liquid within the scope of independent claim 11 as granted and claim amendments have been made with this in mind.

It is clear however that the capsules of Examples 1-5 do not include fragrance, or enzyme, of relevance to the present invention. It is also clear that there is no general teaching or disclosure of the encapsulates useful in the laundry liquids or liquid detergent compositions of the presently claimed invention. Thus, the document includes no disclosure of laundry liquids comprising at least 5% by weight of surfactant, and an encapsulate comprising fragrance in a hydrated cross-linked anionic gum matrix, or an encapsulate comprising enzyme in a hydrated cross-linked anionic gum matrix. Neither is there any teaching or suggestion in D5 of liquid detergent compositions comprising at least 5% by weight of surfactant and an encapsulate comprising greater than 10% by weight of the encapsulate of enzyme or active material comprising a fragrance, in a hydrated cross-linked anionic gum matrix. The subject matter of present independent claims 1, 10, 13 and 14 is therefore novel in view of this document, as is independent claim 9 which is directed to a liquid detergent composition comprising at least 5% by weight of surfactant and an encapsulate comprising greater than 0.5% by weight of the encapsulate of fragrance, in a hydrated cross-linked anionic gum matrix.

We will now turn to the inventive step of the amended claims.

The problem addressed by the present invention is the provision of an encapsulate containing fragrance or enzyme, which can be incorporated in a liquid detergent composition, particularly a laundry liquid, is stable on storage yet dissolves on dilution of the composition (or product), releasing the fragrance or enzyme.

It is acknowledged in the patent specification at paragraph No. 0004 that there is a large amount of literature in the area of fragrance encapsulation, e.g. to provide fragrance stability. It is also known to encapsulate enzymes to protect them, for example, from de-activation by exposure to other ingredients typically present in a detergent composition.

The present invention is based on the appreciation that the use of an encapsulate including a hydrated cross-linked anionic gum matrix to contain the fragrance or enzyme, enables the preparation of encapsulates which are stable *per se* in many liquid product forms and compositions, particularly in aqueous media alone, i.e. water, yet which upon dilution are dissolved by the action of diluted surfactant and/or builder salts which strip out the multivalent catians used to cross-link the matrix.

We consider D1 to be closest prior art for the purposes of inventive step.

D1 addresses a similar problem to that addressed by the patent, namely D1 is directed to the provision of liquid detergent compositions comprising surfactant and encapsulated material, in which the encapsulates are stable during storage yet are able to disintegrate when the composition is diluted with water, thereby releasing the encapsulated material.

D1 discloses that it is desirable in some circumstances to include an electrolyte in the liquid phase of the composition. An inspection of the exemplified compositions of D1 reveals that all of the compositions contain electrolyte, e.g. sodium chloride, triethanolamine, potassium chloride. The presence of an appropriate quantity of electrolyte in the compositions is taught in D1 as possibly stabilising the particles against interaction with surfactant in the liquid phase. By contrast, as discussed above, encapsulates of the presently claimed invention are stable in many product forms and compositions in the absence of electrolyte.

Encapsulates having a matrix structure were found by the present inventors to be more stable than capsules having a wall structure in the claimed compositions. There is no teaching or direction in D1 to select a matrix form of an encapsulate. The examples of D1 teach that it is desirable to use a wall structure for the particles.

Neither is there any teaching or direction derivable from D1 to a person of ordinary skill the art to select perfume as an additional ingredient for the particles. Although perfume is mentioned in D1 as a possible ingredient, this disclosure is in the context that the liquid phase or the particles can additionally comprise perfumes, amongst other ingredients (see page 2, column 2, lines 95-99). It therefore does not necessarily follow that the encapsulates comprise perfume. A further possibility within the disclosure is the absence of any perfume.

In any case, in practice, if perfume were to be employed in the particles of D1, a person of ordinary skill in the art following the experimental method disclosed in the document would find that they would not obtain stable perfume-containing encapsulates. It is preferred in D1 (see page 2, column 1, lines 56-61 and Examples 1 and 2) that when carrageenan is used as the particle material, the particles including carrageenan are hardened by a hardening agent, which preferably comprises a solution containing 1-2.5% by weight of potassium chloride in a 3:1 mixture by weight of ethanol and water. However, ethanol is not an appropriate cross-linking agent for use with fragrance, as it would extract the fragrance from the particles. Accordingly, because ethanol is incompatible with fragrance, the teaching of D1 would not lead a person of ordinary skill in the art to adopt this approach for the preparation of fragrance-containing encapsulates. Therefore, it is clear that the Opponent's conclusion that the use of fragrance/perfume in the encapsulates cannot be regarded as inventive in view of D1 is neither correct, nor appropriate.

Plainly there is no teaching in D1 of enzymes as possible additional ingredients.

There is therefore no teaching derivable from D1 to combine the features of the compositions or liquids as defined in the presently claimed invention. In particular, there is no direction in D1 to select a matrix encapsulate form and, moreover having done so, to then include perfume or enzyme in the matrix encapsulate. We therefore do not consider that the prior art would motivate a person of ordinary skill in the art to formulate a composition or laundry liquid falling within the scope of the present claims as amended. With this in mind, a person of ordinary skill in the art would certainly not arrive a composition within the scope of claims 1, 9 or 13 in combination with claim 8 or laundry liquid within the scope of claims 10 or 14 in combination with claim 8, by consideration of the paper by Guisley K.B. Enzyme Microb. Technol. 1989, vol. 11, November (D6), which simply teaches the properties of various polysaccharides.

The present invention therefore involves an inventive step over D1, whether or not considered together with D6.

As described above, D3 does not disclose encapsulates useful in the compositions or laundry liquids of the amended claims submitted herewith.

There is no mention of perfume or enzymes as possible encapsulate contents.

There is also no explicit teaching or direction in D3 to incorporate the encapsulates of D3 in a liquid detergent composition/laundry liquid comprising a surfactant. The detergent compositions specifically contemplated in D3 are dry products. As discussed above in relation to D1, we believe that the term 'laundry liquid' would be understood by a person of ordinary skill in the art to refer to the products as such, and therefore the products as sold. Similar considerations apply to the term 'liquid detergent composition'. For example, paragraph No. 0047 of the patent specification states that the encapsulates remain intact in the laundry liquid over prolonged storage and retain most of the active material. However, once the product is diluted, the surfactant becomes more soluble and works in combination with the builder to dissolve the encapsulate releasing the active material. In this connection, dilution of the particulate detergent composition in water in the Examples of D3, to form an aqueous wash solution would not constitute liquid detergent compositions or laundry liquids within the scope of the claims.

Moreover, it is well known that different considerations apply when formulating dry products containing capsules as compared with liquid detergents. It is very difficult to formulate liquid detergent compositions containing stable encapsulates. The liquid phase of the detergent composition is likely to attack the encapsulating material and does, in any event, tend to present a more rigorous chemical and physical environment from which the encapsulating material (in the present case an anionic gum) needs protect its contents. It is therefore not appropriate to assume that the encapsulates of D3 would necessarily be stable and remain intact in liquid detergent compositions or laundry liquids containing a surfactant. For this reason, a person of ordinary skill in the art would not from a reading of D3 derive liquid detergent compositions or laundry liquids falling within the scope of the present claims.

As for the Opponent's arguments concerning fragrance, as described at paragraph No. 0062 of the patent, comparative tests have shown that a given amount of fragrance material in a detergent composition according to the present invention produces a stronger fragrance effect, thus providing possibilities for reducing fragrance material usage in product, with consequential cost savings.

The presently claimed invention therefore involves an inventive step over D3.

If the Opposition Division is not minded to maintain the patent on the basis of the amended claims enclosed herewith, we request appointment of Oral Proceedings.

We enclose a copy of this letter and enclosures for the Opponent.

Yours faithfully
KEITH W NASH & CO

MATTHEWS, Heather Clare
Authorised Representative

Encs.

C281.00/Q

EP 00901233.7

AMENDED CLAIMS

1. A liquid detergent composition comprising at least 5% by weight of surfactant and an encapsulate comprising greater than 10% by weight of the encapsulate of active material comprising fragrance, in a hydrated cross-linked anionic gum matrix.

2. A detergent composition according to claim 1, wherein the surfactant is present at a concentration in the range from 5 to 50% by weight.

3. A detergent composition according to claim 2, wherein the surfactant is present at a concentration in the range from 10 to 30% by weight.

4. A detergent composition according to any one of the preceding claims, wherein the surfactant is selected from the group consisting of anionic, nonionic, amphoteric and zwitterionic surfactants and mixtures thereof.

5. A detergent composition according to any one of the preceding claims, wherein the fragrance is present in an amount up to 60%, preferably in the range from 20 to 40%, and particularly 25 to 30% by weight of the encapsulate.

6. A detergent composition according to any one of the preceding claims, wherein the anionic gum is present in an amount up to 5%, preferably up to 1% by weight of the encapsulate.

7. A detergent composition according to any one of the preceding claims, wherein the anionic gum is alginate, carrageenan, gellan gum, carboxymethyl cellulose and/or xanthan gum.

8. A detergent composition according to claim 7, wherein the anionic gum is alginate.

9. A liquid detergent composition comprising at least 5% by weight of surfactant and an encapsulate comprising greater than 0.5% by weight of the encapsulate of fragrance, in a hydrated cross-linked anionic gum matrix.

10. A laundry liquid comprising at least 5% by weight of surfactant and an encapsulate comprising fragrance in a hydrated cross-linked anionic gum matrix.

11. A laundry liquid according to claim 10, wherein the liquid comprises in the range from 5 to 50% by weight of anionic surfactant.

12. A laundry liquid according to claims 10 or 11, wherein the liquid additionally comprises in the range from 5 to 80% by weight of builder.

13. A liquid detergent composition comprising at least 5% by weight of surfactant and an encapsulate comprising greater than 10% by weight of the encapsulate of enzyme, in a hydrated cross-linked anionic gum matrix.

14. A laundry liquid comprising at least 5% by weight of surfactant and an encapsulate comprising enzyme in a hydrated cross-linked anionic gum matrix.

www.nyscc.org/news/archive/tech0202.htm

Tech Notes

Encapsulation: New Tools for Ingredient Delivery

By Dallas Hetherington, National Starch and Chemical Company

I look at skin creams and cans of hair s pray, the items that we see every day on the supermarket shelves, as delivery systems. Why? Their purpose is to deliver specific ingredients to the skin or hair. But today, the term delivery system is accepted to mean something different. To most people, these examples do not qualify as delivery systems because they are well known, traditional delivery tools. Instead, when one hears the term delivery system, one thinks of newer systems and tools such as transdermal patches or, the topic of this article,: encapsulation.

Encapsulation has become increasingly important in personal care to aid in the protection, delivery and release (yes, even controlled release!) of actives, particularly to the skin, in a variety of applications. Actives for encapsulation include moieties like self tanning agents; sunscreens; and specialty ingredients such as vitamins; skin bleaches, anti-acne agents, and anti-aging treatments.

The major categories of encapsulation are differentiated principally by size and include nano-particles; micro-capsules and macro-capsules (some say milli-capsules). Examples of these encapsulate varieties are liposomes, which are measured in terms of nanometers; true "core/shell" particles which are measured in microns; and larger particulates which are plainly visible to the naked eye and can be measured in millimeters.

The functionality of encapsulates can be dependent on their particle size. In skin care, there is much focus on nano-particles as delivery systems because these can be used to help usher actives into the skin layer itself. Their small size enables penetration into the stratum corneum. In contrast, macro-capsules provide mainly a visual or marketing effect because they can be easily seen within a clear formulation. Because of their large size, macro-capsules can be more easily engineered to protect the ingredient inside, as the shell, or outside, protective wall is easier to form and make uniform.

Micro-capsule technology is a field in which there are many application possibilities. With micro-capsules, materials can be protected either by a core/shell structure, where the core is the active you wish to protect, or by entrapping the active in a matrix structure. The core/shell model is easy to visualize - just think of a candy like an M&M - where the core is the peanut and the shell is the chocolate coating. To visualize the entrapment/matrix model, you can imagine a sponge where the active is entrapped in the voids inside the sponge. In this case, some of the active can be seen from the outside.

Globally, one of the most commonly used methods of encapsulation is specialty starch-based encapsulation following the entrapment/matrix model described above. This technology is used frequently in the food industry to encapsulate flavors for drink and sauce mixes. The technology is also used for the encapsulation of fragrances for household products, and is used today to encapsulate vitamins for healthcare applications.

Starch matrix encapsulation technologies have a number of interesting applications for personal

care. A starch matrix can provide additional stability to a fragrance. Fragrances are volatile and can escape from a finished product over time. They are also susceptible to oxidation. Starch encapsulation can address both of these issues. Other skin care ingredients have been successfully encapsulated in specialty starches, including petrolatum, a variety of silicone based materials, and other products. The amount of active that can be entrapped as a percentage of the total weight of the encapsulate or final particle can be as high as 70-80%, while still maintaining a free flowing powder.

Starch encapsulates can be designed to optimize ingredient delivery. The particles are often engineered to be trigger-released via contact with moisture, such as in a bath powder product or in a deodorant where body moisture triggers the release of a fragrance. Starch materials are also used as coatings for other particles and can be designed to release quickly or slowly in water, depending upon need. Specialty starches can be selected to deliver optimized aesthetic properties, as well as optimized delivery properties.

The majority of encapsulation today, including starch-based encapsulation, takes place at third party organizations. These are often the manufacturers of active ingredients who then sell the encapsulated materials to the formulators of consumer products. However, as the value of encapsulation as a delivery system becomes more widely recognized, an increasing number of formulating companies are working directly with suppliers of the specialty encapsulating ingredients in order to foster new innovations.

Some day, I suspect that the cosmetic chemist will view nano-particle, macro-capsule, and even micro-capsule delivery technologies the same way we look at skin creams and cans of hair spray today. However, based on the extent of ever-emerging technologies and possibilities for encapsulation, this day is likely far off in the distant future.

Other vintage New York SCC Tech Articles can be read online [more].

ACS SYMPOSIUM SERIES 520

Polymeric Delivery Systems

Properties and Applications

Magda A. El-Nokaly, EDITOR
The Procter & Gamble Company

David M. Piatt, EDITOR
The Procter & Gamble Company

Bonnie A. Charpentier, EDITOR
Syntex Research

Developed from a symposium sponsored
by the Division of Cellulose, Paper and Textile Chemistry
and the Biotechnology Secretariat
at the 203rd National Meeting
of the American Chemical Society,
San Francisco, California,
April 5–10, 1992

American Chemical Society, Washington, DC 1993

any polymeric delivery
ialized polymeric drug
des such as biodegrad-
Polymeric Delivery Sys-
on polymeric delivery
xchange, dialogue, and

on of Cellulose, Paper
retariat for sponsoring
er & Gamble Company
are due to Procter &
Office; Ken Smith and
sion; Robert Boggs and
n; and Lynda Sanders

for their cooperation,
. Many thanks to the
cially Anne Wilson, for
wledge with thanks the

Chapter 1

Polymer Delivery Systems Concepts

I. C. Jacobs and N. S. Mason

Department of Chemical Engineering, Washington University, One Brookings Drive, St. Louis, MO 63130

Examples of successful applications of controlled release are mentioned and commonly used concepts are defined. The chapter describes how polymers are used in devices and how their properties affect device performance. Processes of wide applicability including those covered in succeeding chapters are summarized.

The task of controlled release is deceptively simple: get the right amount of the active agent at the right time to the right place. Controlled release is a term that represents an increasing number of techniques by which active chemicals are made available to a specified target at a rate and duration designed to accomplish an intended effect. In their most elegant implementation, these systems can mimic processes of living cells such as secretion of hormones or enzymes. Many other terms besides "controlled" have been used to describe somewhat different delivery system concepts from "continuous" release to "timed" release. Some of the terms are defined more precisely by Ballard (*1*). Only a few examples will be cited in the paragraphs following.

Controlled Release of Drugs. Controlled release is often used to extend the time the effective therapeutic dose is present at the target from a single administration, and to avoid or minimize concentrations that exceed therapeutic requirements. It also can decrease the needed dose of an expensive active ingredient. Protecting certain tissues is often desirable, e.g. the stomach from irritation. Targeting tissues may be desirable, avoiding toxic effects. This can make the administration of the drug less invasive. Taste-masking of a drug can improve patient compliance. Incompatibilities between ingredients can be mitigated. A few commercial examples may be mentioned: Occusert, a device for releasing pilocarpine, a drug to treat glaucoma, to the eye (*2,3*), an implantable osmotic pump capable of delivering a nearly constant 20 mg/hr of solution to the body for almost a day (*4*) and Norplant, a long-term contraceptive implant (*5*).

Controlled Release Pesticides. Controlled release can increase the effectiveness of an agent, and its specificity. It can decrease the possibility of damage to the environment.

0097–6156/93/0520–0001$06.00/0

Penncap M and E, represent examples of microencapsulated forms of methyl and ethyl Parathion (*6*). Systems have been developed for releasing pheromones to confuse insects (*7*), collars to keep fleas away from pets (*8*), even keep mollusks from the hulls of ships (*8*).

Controlled Release Fertilizers. Controlled release can decrease the number of applications. It can make one application last longer. A reduction in run-off into rivers or aquifers may be another benefit.

Special Control Release Applications. "Carbonless carbon paper" was the first product involving microencapsulation. A latent dye, crystal violet lactone is contained in 5 to 25 micron microcapsules that are coated on the back of a page. The action of a sharp pencil, typewriter, or impact printer ruptures the microcapsules, releasing the lactone to react with an acid clay present on the copy sheet, activating the color (*9*).

Side effects in the treatment of diabetes would be decreased, if encapsulated implants of islets of Langerhans from the pancreas could be used. The islets could originate from a different species. Encapsulation would keep the islets from being destroyed by the immune system of the host, allowing the cells to release insulin in response to the concentration of glucose (*10*).

Improvements in oil well stimulation with hydraulic fracturing fluids have been realized with the use of controlled release viscosity breakers (*11*). Increasing the likelihood of germination of seeds under adverse conditions is another application of controlled release. Because controlled release is a rapidly changing field, new ideas are being generated and applications are being evaluated every day.

Functions of Polymers in Controlled Release.

Polymers are uniquely suited as materials of construction for delivery systems because their permeability can be modified and controlled. They can be shaped and applied relatively easily by a large variety of methods. Active ingredients and property modifiers can be incorporated either physically or chemically. In general, polymers have little or no toxicity. Despite the diversity of applications, they principally serve as membranes or envelopes, as matrices in which the active ingredient is dispersed or dissolved, or as carriers which are chemically attached to the active ingredient. Not all controlled release devices use polymers explicitely. For example liposomes do not.

Delivery Systems Terms.

Devices can range in size from as small as one molecule to coated tablets and boluses used in cattle.

Availability, or bioavailability is an important property of most drug-containing devices. Tests in glass-ware (*in vitro*) or in the living system (*in vivo*) must be conducted to ascertain that the active ingredient is available under specified conditions.

Biodegradable refe
molecular weight ev
applied to polymers
poly(ε-caprolactone)
enzymes are involve
insensitive to pH. N
and enzymes are inv
proteins have also b
biodegradable were,
polyurethanes, and p

Biocompatible devi
effects in living syste

Bioabsorbable devi
utilized or metaboliz

Erodible systems ar
of the matrix.

Zero order release n
analogy to reaction k
reservoir device cont
rate-limiting membra
device and a non-ch

First order or pseud
concentration remair
decrease in rate with
concentration decrea

Burst effect refers t
more rapidly during
steady-state, or a late

Time lag is observed
concentration gradie
elapses before the rel
this and the above pl

Reservoir devices c
controlling membran
where the wall of the
substance coated wit

Biodegradable refers to polymers that under certain conditions undergo a decrease in molecular weight eventually disintegrating or dissolving in the medium. It is most often applied to polymers and copolymers of lactic, glycolic, hydroxybutyric acids as well as poly(ε-caprolactone). For these substances the degradation is hydrolytic and no enzymes are involved. Kinetics generally follow first order (*12, 13*) and are often insensitive to pH. Natural polymers such as starch and cellulose are also biodegradable and enzymes are involved in these cases. Polyphosphazenes, polypeptides, and proteins have also been proposed for drug delivery. Other polymers mentioned as biodegradable were, poly(dihydropyrans), poly(acetals), poly(anhydrides) (*14*), polyurethanes, and poly(dioxinones).

Biocompatible devices are devices that can be applied without causing undesirable effects in living systems.

Bioabsorbable devices are those which are degraded by a living system and can be utilized or metabolized by it.

Erodible systems are designed to control the release of the active substance by erosion of the matrix.

Zero order release means the active substance is released at a constant rate. By analogy to reaction kinetics, the rate of release, $-dc/dt = k$. It can be approximated by a reservoir device containing a saturated solution of the active ingredient surrounded by a rate-limiting membrane. This requires a supply of undissolved active ingredient in the device and a non-changing or zero sink condition (*15*).

First order or pseudo-first order means the release rate is proportional to the concentration remaining in the device i.e., with a rate, $-dc/dt = kc$. This implies a decrease in rate with time. It would be approximated by a device in which the concentration decreased as the amount remaining in the device decreased.

Burst effect refers to the tendency of some devices to release the active substance more rapidly during some period, usually the initial test period, than during the steady-state, or a later period.

Time lag is observed when the rate limiting membrane must first establish a concentration gradient before releasing at the designed rate. Therefore some time elapses before the release rate reaches its designed value. Mathematical expressions of this and the above phenomenon are given in (*15*).

Reservoir devices consist of a drug or other active agent enclosed within an inert controlling membrane. Examples would be a tube filled with the active substance, where the wall of the tube would serve as the limiting membrane, a sphere of the active substance coated with a film controlling the diffusion of the active substance, or a slab

of the active substance closed off from the medium by a film which controls the diffusion.

Matrix devices in which the active drug is dispersed throughout the polymer are called monolithic devices or monoliths by a number of authors. One can distinguish between monolithic devices in which the active ingredient is dissolved, dispersed, located in connected pores, or granular. The equations describing the time behavior of such devices predict that initially the fraction released varies as $time^{1/2}$ and the rate of release is proportional to $time^{-1/2}$. Baker and Lonsdale (*15*) in their classic paper, treat each of the cases separately including the different geometries such as slab, sphere and cylinders. As time increases, the release rate is better approximated by an exponential decay (Late time approximation). A more recent review of this material by Roseman may be found in (*16*).

Targeting of drugs is a concept that is as old as the dream of the magic bullet of Paul Ehrlich (*17*). Thies (*18*) distinguishes between active and passive targeting. Passive targeting takes advantage of an existing body process such as the rapid concentration of particles smaller than 1 micron in the liver or spleen after intravenous injection. In active targeting, the natural tendency of the body to distribute the active substance is altered. The drug or carrier, because it has a special affinity e.g., by certain molecular interactions such as a lock and key mechanism, interacts with specific cells. If this could be implemented, it could improve cancer chemotherapy considerably.

Enteric coatings, coatings which are less soluble in acidic aqueous solutions than neutral ones, provide a means for a drug to by-pass the stomach and only become available in the intestine. This is a method to direct rather than target a drug. Many enteric coatings are based on polymers containing phthalic acid residues attached to cellulosic or vinyl polymer chains. Reverse enterics, on the other hand, do not dissolve in neutral media such as the mouth, but dissolve very rapidly in an acid medium such as the stomach.

Polymer Properties that Affect the Release of Active Substances.

Consider a polymer membrane and a diffusing active substance in solution. The amount of the substance which diffuses per unit time across the membrane at steady-state is proportional to the diffusion coefficient and to the concentration difference across the membrane measured on each side of the membrane just inside the membrane. (It is also proportional to the area and inversely proportional to the thickness of the membrane). Because the concentration inside the membrane is not known and is often much different from the concentration in solution, the concentration difference normally measured in the solution must be multiplied by the distribution coefficient. The distribution coefficient is the ratio of the solubility of the agent in the membrane to its solubility in the external medium. The permeability is the

product of the diffusion
same as the diffusion c

It must be pointed out,
diffusivity and solubility
attainable with delivery
silicone rubbers which
active molecules of inte
mg/cm^2 through a 0.1 m
orders of magnitude lo
favored silicone rubbers
these parameters are list
The diffusion coefficien
different dependencies i

Tabl

Solute
Acetophenone
Chlormadione Acetate
Estriol
Fluphenazine
Hydrocortisone
17α-hydroxy-progesteron
Progesterone
Salicylic Acid

SOURCE: Adapted from

Hydrophilic polymers, or
subject to the limitations
in a later section. Porous